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Environment-friendly Electrolyte to Optimize the Surface Properties of Nitinol Tubes

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Nitinol are widely used in biomedicine, mechanical electronics, daily products, and petrochemicals because of their excellent mechanical properties, high-fatigue resistance, corrosion and wear resistance, and biocompatibility. To improve the surface integrity and biocompatibility of nitinol tubes, a polishing method based on an environment-friendly electropolishing solution was studied. The influence of the electropolishing parameters on the surface quality and the degrees of influence were evaluated using orthogonal and single-factor experiments. The removal of material and wettability of the electropolishing process were also analyzed quantitatively. Results showed the good quality of the polished nitinol tube under the optimal polishing parameters. In addition, the amount of material removed was controlled, and biocompatibility was enhanced.

Keywords: Environment-friendly electrolyte; Nitinol tube; Surface properties; Ra; Electrolytic polishing; SEM

1. INTRODUCTION

Nitinol is a type of functional material discovered in the 1970s [1]. This alloy has excellent mechanical properties, desirable service performance, biocompatibility, high-fatigue resistance, and corrosion and wear resistance. Thus, this material is widely used in biomedicine, mechatronics, daily products, and petrochemicals [2–4]. Given the progressing global economy, cardiovascular diseases have seriously threatened human life and health worldwide [5]. These diseases have high prevalence, high disability, and high mortality, recently ranking first in mortality caused by diseases [6,7]. Interventional therapy has become the main treatment for cardiovascular diseases because of its advantages, such as low damage, high safety, and obvious effects [8,9]. Nitinol with excellent properties are widely applied in interventional therapy. At present, a nitinol medical equipment mainly includes laser processing and EDM [10,11]. However, the processed nitinol tube has thermal damage, heat-affected zone, recast layer, and poor surface integrity [12–14]. These defects can cause problems, such as thrombosis, carcinogenesis, and inflammation, in the human body [15]. Electropolishing is a good technique to resolve these defects. This process can reduce the surface roughness (Ra) of the material, improve its corrosion resistance and friction resistance, and finally obtain a smooth, bright anode. It has a minimal damage to the workpiece and a high polishing efficiency. It is a common method for the surface treatment of the workpiece in production [16–18].

Rokicki et al. studied the effect of sodium hypochlorite electrolyte on the surface properties of nitinol metal plates and increased the oxide layer on the surface of the alloy [19]. Ji et al. used an acidic electrolyte, with perchloric acid as the main component, to electrolytically polish the nitinol tube. This electrolyte reduced the surface roughness (Ra) but improved the surface morphology of the tube [20]. Simka et al. compared the effects of three acidic electrolytes on the surface quality of nitinol metal plates. The surface of the polished alloy showed better surface performance in the electrolyte mainly composed of hydrofluoric and sulfuric acids [21]. Neelakantan et al. studied the mechanism of polishing nitinol metal plates in an electrolyte mainly composed of sulfuric acid. Given that the parameters were not optimized, after electropolishing, although a salt film formed on the surface of the alloy, the Ra increased by two-fold [22]. Wang et al. combined glacial acetic acid and perchloric acid as the main solution to electrolytically polish the nitinol tube, and they determined the best electropolishing parameters and improved the surface morphology of the tube [23].

Current studies on nitinol have mainly focused on nitinol plates. With distinct structures, the parameters for optimizing the surface properties of these alloys obviously vary. The only research on nitinol tubes used mainly acidic electropolishing solutions, which are not environment-friendly, and the polishing process was difficult [20,23]. The present study consisted of four parts. First, the nitinol tube was used as the research object. Second, the electrolyte ratio was determined using an environment-friendly solution as the main constituent and a certain amount of additives were added. Third, the effects of four polishing parameters on the nitinol tube were analyzed and optimized. Finally, the surface morphology, Ra, wettability, and wall thickness of the tubes before and after polishing were evaluated.

2. EXPERIMENTAL

A nitinol tube (Ni 52.3at%, Ti 47.7at%, Jiu Steel Group) was used as the workpiece. The tube had an outer diameter of 2.65 mm and wall thickness of 0.24 mm. An LF85 tube fiber laser cutting machine was used to cut the tube into pieces with length of 11 mm. The cut workpiece was ultrasonically cleaned in absolute ethanol for 45 s, dried, and then polished with 600#–1000# sandpaper to remove the residual large slag and oxides on the surface of the workpiece. After polishing, the tube was cleaned again and put in the drying box to dry.

An optical profiler (MicroXAM-100, Shanghai Micro-Nano Metrology Technology Co., Ltd., China) was used to measure the Ra of the nitinol tube. Three different positions on the workpiece were selected, and each position was inspected thrice. The average of the measured Ra values was calculated to be 1.37 μm. A field-emission scanning electron microscope was used to determine the surface morphology of the workpiece (Figure 1). Numerous dents, slag, and oxide were found on the surface of the workpiece. Figure 1 shows that the surface topography of the workpiece was poor, and the surface flatness needed further improved.

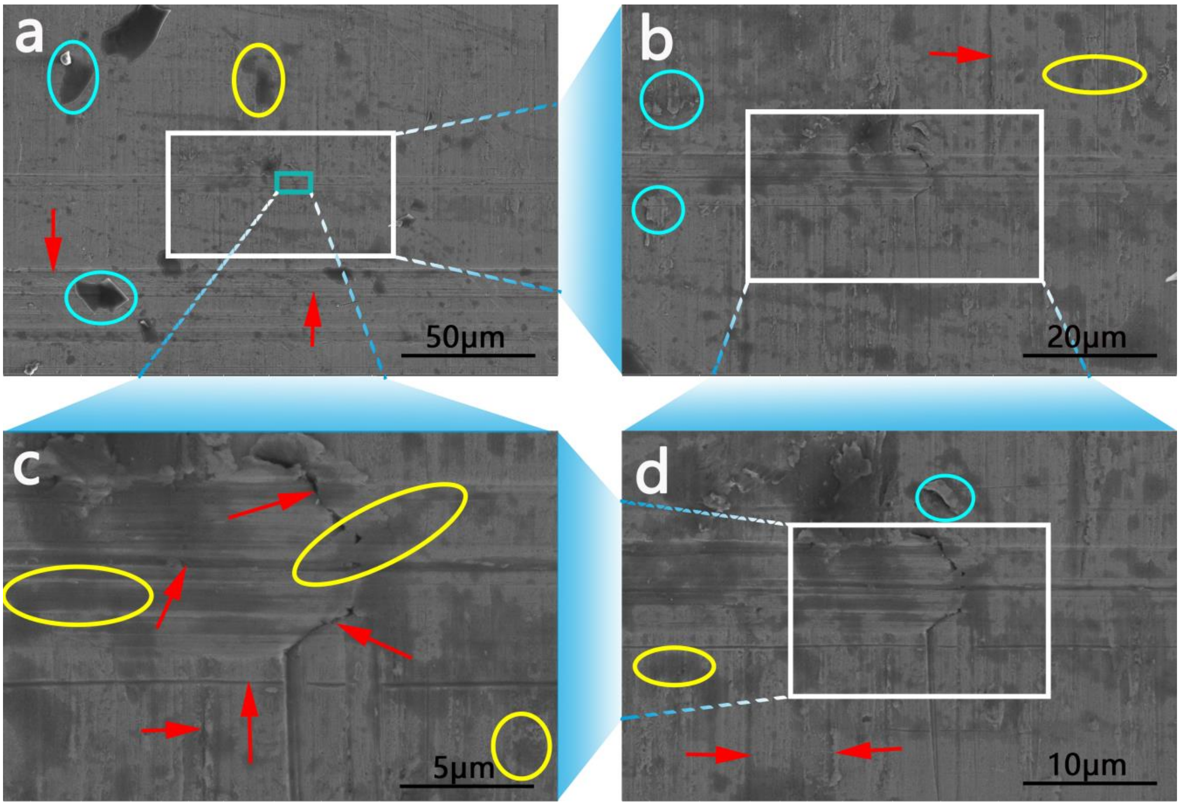

Figure 1. Original surface micromorphology of the nitinol tube: (a) low magnification; (b) partially enlarged view of (a); (c) partial enlarged view of (a) and (d); (d) partial enlarged view of (b)

Electropolishing is a method for the anodic treatment of metals by electrolysis to improve the metal surface [24]. The schematic diagram of an electropolishing system is shown in Figure 2. The system was mainly composed of a magnetic stirrer, a digital multimeter, a DC power supply, a fixture, an anode, and a cathode. The magnetic stirrer controlled the speed and temperature of the electrolyte. The composition and polishing parameters of the electrolyte are shown in Table 1.

The wall thickness of the workpiece was measured using an optical profiler. Four different positions on each tube were measured, and each position was measured thrice. The average value was calculated. The material removal rate was determined by dividing the average reduction in the wall thickness by the polishing time.

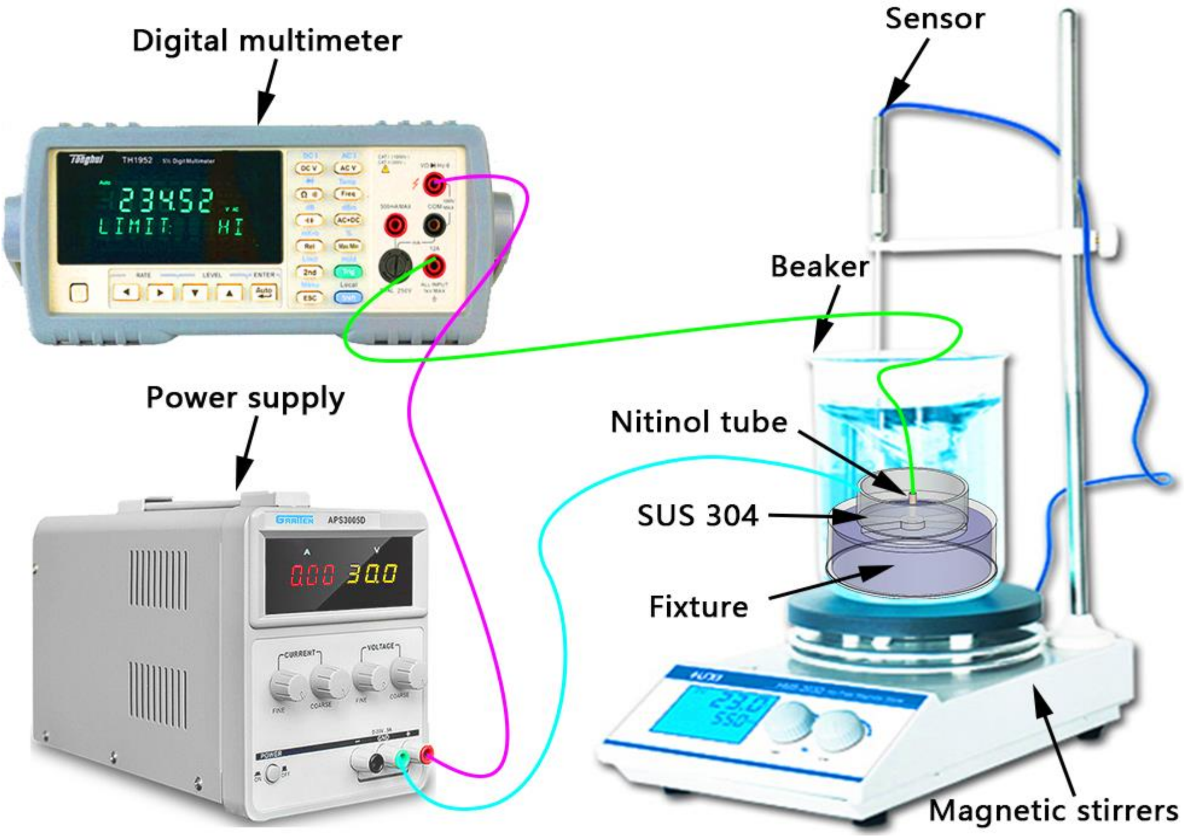

Figure 2. Schematic diagram of the electrolytic polishing system platform

Table 1. Composition and polishing parameters of the electrolyte

Electrolyte A	Additive (ml/L)	Polishing time (min)	Distance between electrodes (mm)	Voltage for polishing (V)	Electrolyte temperature (°C)
1mol/L sodium chloride-ethylene glycol (70% volume fraction) and ethanol (30% volume fraction)	Calcium chloride solution 10-50	3-19	6-26	8-35	14-36

3. RESULTS AND DISCUSSION

3.1 Optimization of the electropolishing solution

The composition of the electropolishing solution is an important factor that affects the polishing of a material. An ideal electrolyte can accelerate the polishing process and effectively reduce the Ra. A smooth and flat polished surface improves the surface integrity [25]. The high viscosity of the polishing liquid will inhibit the diffusion of the anode-dissolved products into the polishing liquid. The gas generated during the hydrogen evolution reaction in the polishing tank isolates the polishing liquid from the metal surface, and the solution cannot be replenished to the anode in time. This phenomenon reduces the dissolution rate of the metal. Otherwise, too-low viscosity will accelerate the diffusion of the dissolved products into the polishing liquid. This phenomenon increases the dissolution rate and affects the surface integrity of the anode workpiece. Given the relatively high viscosity of electrolyte A, different concentrations of calcium chloride were added to the electrolyte to reduce its viscosity. According to the preliminary experiments, the initial electropolishing parameters were as follows: electrolyte A, calcium chloride solution, 10–50 mL/L; voltage for polishing, 21 V; distance between electrodes, 16 mm; electrolyte temperature, 24 °C; and polishing time, 12 min. The Ra and micromorphology of the tube were used as the indices of the polishing parameters. To ensure accuracy, each group of variables was subjected to parallel experiments with four workpieces. After the experiment, an optical profiler was used to measure the Ra and residual wall thickness of the stent (Figure 3).

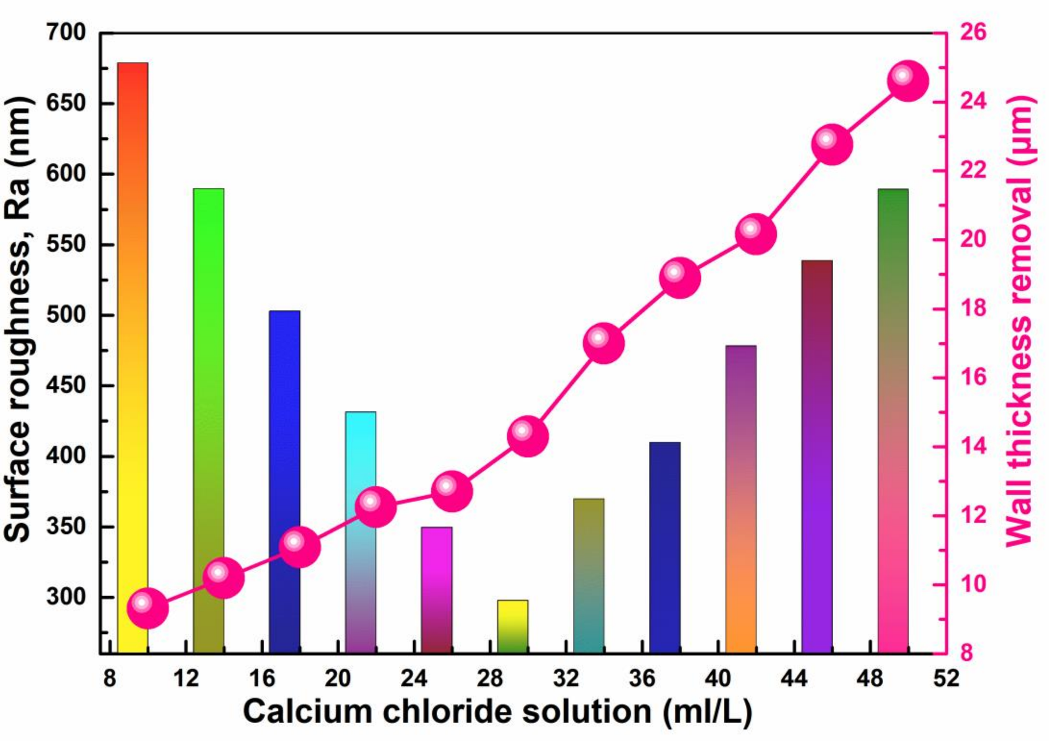

Figure 3. Effect of the concentration of calcium chloride solution (10–50 mL/L) on the surface roughness (Ra) and wall thickness

Figure 3 shows that as the concentration of calcium chloride was increased, the Ra of the nitinol tube first decreased and then increased. When 10 mL/L calcium chloride was added, the Ra of the tube was 678.8 nm, indicating a reduction by 50.45%. Further increase in the concentration of calcium chloride resulted in a gradual decrease in Ra. When the calcium chloride added was 30 mL/L, the Ra of the nitinol tube reached the minimum value of 297.8 nm. Further increase in the concentration of calcium chloride resulted in the gradual increase in Ra. Therefore, the smallest Ra of the nitinol tube could be obtained at 30 mL/L calcium chloride. In Figure 3, the right axis indicates the change in wall thickness. As the concentration of calcium chloride increased, the reduction in the wall thickness of the nitinol tube gradually increased. This phenomenon may be due to the fact that adding calcium chloride, which reduced the viscosity of electrolyte A, accelerated the diffusion of the anode-dissolved products into the electropolishing solution and reduced the occurrence of hydrogen evolution reactions. When the minimum roughness was obtained, the wall thickness of the nitinol tube was reduced by 14.3 μm.

3.2 Optimization of electropolishing parameters

As shown by the results the electropolishing parameters in section 3.1, the surface quality of the workpiece needed further enhancement. Therefore, the electropolishing parameters were further improved through an orthogonal experiment. The L_9 orthogonal array was used to determine the relative optimal conditions, thereby reducing time and cost [26]. Three levels of each factor were selected as follows: polishing time, 6, 11, and 16 min; distance between electrodes, 9, 16, and 23 mm; voltage for polishing, 12, 21, and 30 V; and electrolyte temperature, 16 °C, 21 °C, and 36 °C. The factors and levels of the orthogonal experiment are shown in Table 2.

Table 2. Factors and levels used in the orthogonal experimental design

Level	**Polishing time (min)**	**Distance between electrodes (mm)**	**Voltage for polishing (V)**	**Electrolyte temperature (°C)**
1	6	9	12	16
2	11	16	21	21
3	16	23	30	36

The test results for Ra under the orthogonal design conditions are shown in Table 3. The order by which the experiments were performed was randomized to avoid any kind of bias. All results at each step were expressed as the average of three experiments. Table 4 shows the evaluation indicators of the orthogonal experiment, in which K_i (I = 1, 2, 3) and R were important parameters. K_i was the average value of the Ra of the jth (j = 1, 2, 3) level corresponding to the factor f (f = polishing time, distance between electrodes, voltage for polishing, or electrolyte temperature). R was the difference between the $K_{\max}$ and $K_{\min}$ values in the corresponding factor column. For example, when the coefficient f = polishing time was 1 (6 min), $K_1 = (548.7+369.5+420.2)/3$, $R = \max\{446.13, 320.03, 387.40\}-\min\{446.13, 320.03, 387.40\}$. The optimal factor level could be obtained by comparing the values of K. R was an important indicator, because it represented the range in which K fluctuated. Therefore, the larger the R value was, the more important the factor would be [27,28].

Table 3. Test plan and roughness results

Test	Polishing time (min)	Distance between electrodes (mm)	Voltage for polishing (V)	Electrolyte temperature (℃)	Surface roughness Ra (nm)
1	1	1	1	1	548.7
2	1	2	2	2	369.5
3	1	3	3	3	420.2
4	2	1	2	3	310.9
5	2	2	3	1	327.8
6	2	3	1	2	321.4
7	3	1	3	2	404.1
8	3	2	1	3	420.2
9	3	3	2	1	337.9

Table 4. Results of roughness analysis

Parameters	Polishing time	Distance between electrodes	Voltage for polishing	Electrolyte temperature
K_1	446.13	421.23	430.10	404.80
K_2	320.03	372.50	339.43	365.00
K_3	387.40	359.83	384.03	383.77
R	126.10	61.40	90.67	39.80

Based on the orthogonal experiments, the best parameters were as follows: polishing time, 11 min; distance between electrodes, 23 mm; voltage for polishing, 21 V; and electrolyte temperature, 21 ℃. The comparative results of the surface morphologies before and after polishing are shown in Figure 4. Compared with those before polishing (Figure 4a), the slag and oxides disappeared from the surface of the nitinol tube after the tube was polished under the abovementioned parameters (Figure 4b), indicating an improved polishing effect. *R* reflects the influence of various factors on the Ra of the nitinol tube. Table 4 shows that polishing time had the greatest influence on Ra, followed by voltage for polishing, distance between electrodes, and electrolyte temperature.

Based on the orthogonal experiment, the optimal values and importance of the electropolishing parameters were determined. However, due to the limitations of the method, the optimal result did not exceed the range of the level considered [29,30]. Therefore, single-factor experiments were performed to further optimize the parameters. All results were expressed as the average of three experiments.

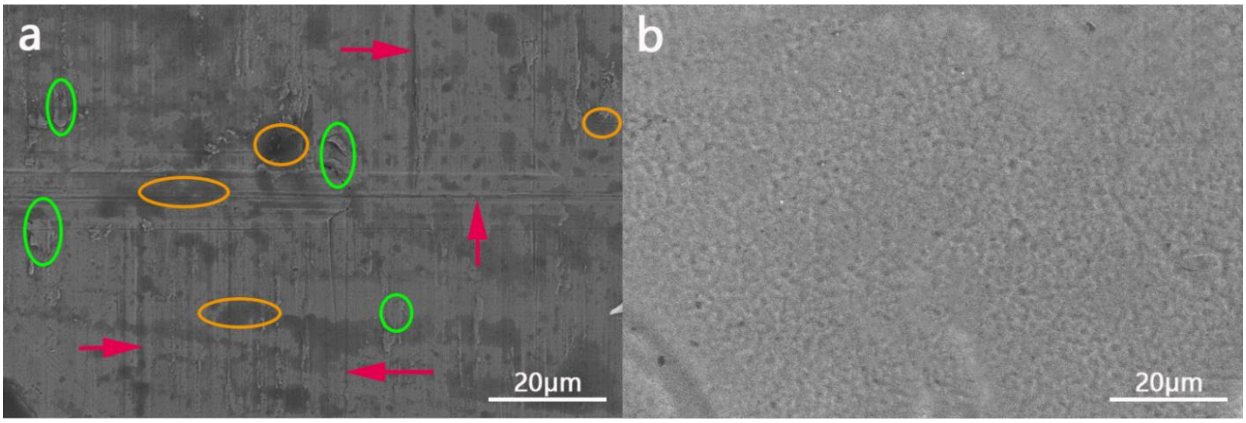

Figure 4. Comparison of the surface micromorphologies of the nitinol tube: (a) before and (b) after the parameters were optimized

3.2.1 Optimization of polishing time for the nitinol tube

After the orthogonal experiment, single-factor experiments were performed to optimize the polishing parameters. The polishing time was used as a variable and varied in the range of 3–19 min. The remaining parameters were based on the results of the orthogonal experiment (voltage for polishing, 21 V; distance between electrodes, 23 mm; and electrolyte temperature 21 °C). The average fitting curve of polishing time versus the Ra values of the polished nitinol tube is shown in Figure 5.

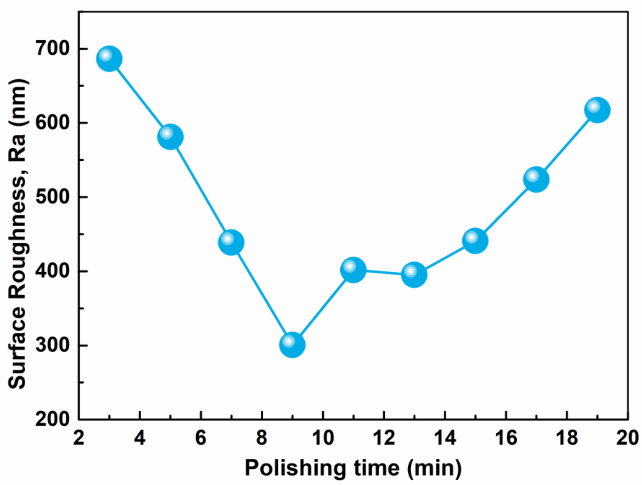

Figure 5. Average fitting curve of polishing time (3–19 min) versus Ra of the nitinol tube

Figure 5 shows a generally V-shaped curve. When the polishing time was 3 min, the minimum, average, and maximum Ra values were 542.69, 686.42, and 846.20 nm. Compared with the optimized parameters from the orthogonal experiment, the surface of the tube became rough instead, because the polishing time was too short, and the surface was not sufficiently polished. When the polishing time was less than 9 min, the Ra of the tube gradually decreased as the polishing time was prolonged. However, when the polishing time was more than 9 min, the Ra of the tube gradually increased. Thus, the minimum Ra (231.25 nm) was obtained with the polishing time of 9 min. The average Ra (300.55 nm) and maximum Ra (354.7 nm) were also optimal at this polishing timepoint. Therefore, the best electropolishing time was 9 min.

3.2.2 Optimization of the polishing voltage for the nitinol tube

Then, the polishing voltage, which was the second most influential parameter, was also optimized. The parameter range of the polishing voltage was set to 8–35 V. According to section 3.2.1, the polishing time was 9 min. The remaining parameters were based from the results of the orthogonal experiments (distance between poles, 23 mm; electrolyte temperature, 21 °C). The average fitting curve of polishing voltage versus the Ra of the polished nitinol tube is shown in Figure 6.

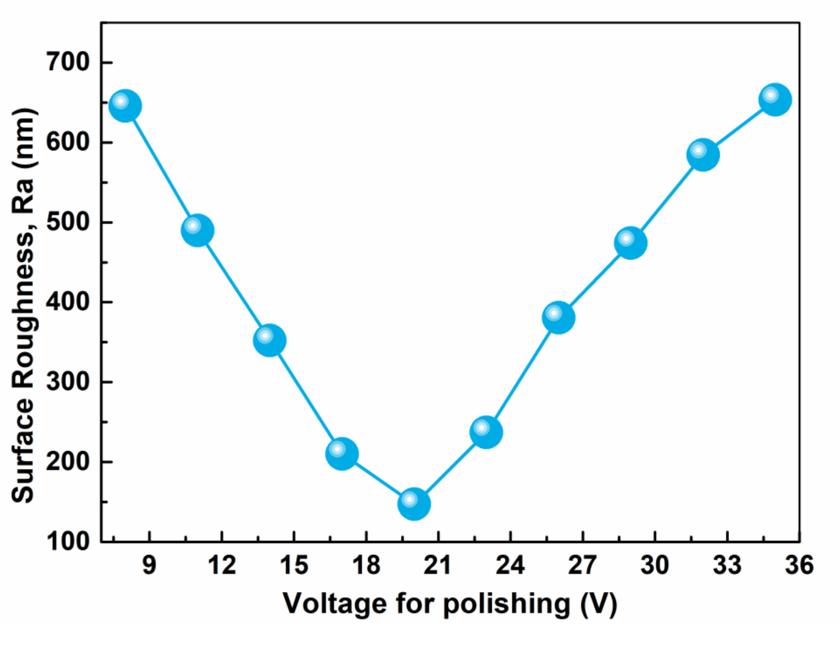

Figure 6. Average fitting curve of the polishing voltage (8–35 V) versus Ra of the nitinol tube

The Ra decreased first and then increased as the polishing voltage increased, and the change pattern was V-shaped. When the polishing voltage was 8 V, the minimum, average, and maximum Ra values were 480.67, 645.70, and 728.86 nm. Given the very small polishing voltage, the original defects on the surface of the nitinol tube were not completely removed, resulting in the increased Ra. At polishing voltage of less than 20 V, the Ra of the tube gradually decreased with prolonged polishing time. When the polishing voltage was above 20 V, the Ra of the tube gradually increased. The minimum roughness (97.64 nm) corresponded to a polishing voltage of 20 V. At this time, the average roughness (147.33 nm) and maximum roughness (203.09 nm) were also at optimal levels. Therefore, the best electropolishing voltage was 20 V.

3.2.3 Optimization of the distance between electrodes for the nitinol tube

According to Table 4, the third important factor for the surface quality of the nitinol tube was the distance between electrodes. Therefore, a single-factor test was also performed on the distance between electrodes. The parameter range of the distance between electrodes was set to 6–26 mm. According to sections 3.2.1 and 3.2.2, the polishing time was 9 min, and the voltage for polishing was 20 V. The electrolyte temperature was 21 °C, according to the result of the orthogonal experiment. The average fitting curve of distance between electrodes versus Ra the polished nickel–titanium alloy tube is shown in Figure 7.

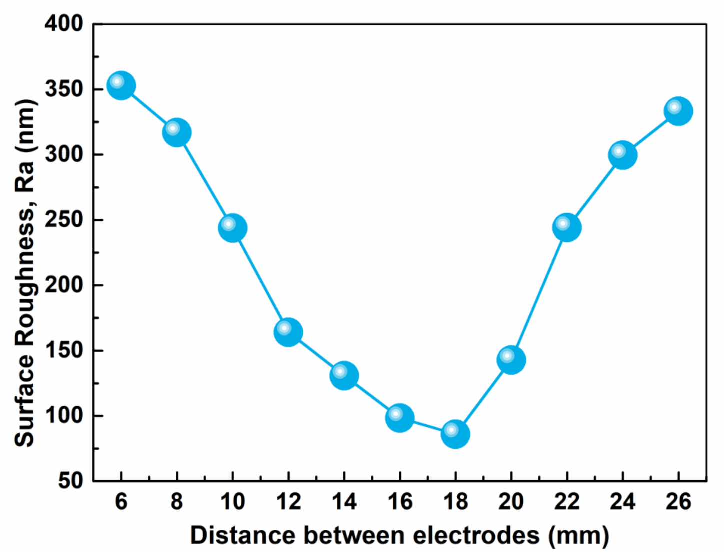

Figure 7. Average fitting curve of the distance between electrodes (6–26 mm) versus Ra of the nitinol tube

The Ra decreased first and then increased as the distance between electrodes was increased. When the distance between electrodes was 6 mm, the minimum, average, and maximum Ra values were 238.93, 352.82, and 452.05 nm. Given that the gap between the electrodes was too small, the solution after electropolishing could not be discharged in time, and the new electrolyte could not be replenished in time. These phenomena resulted in the suppression of subsequent reactions, resulting in low surface quality [31]. When the distance between the electrodes was above 18 mm, the Ra of the tube gradually increased. The minimum Ra (42.7 nm) was determined when the distance between the electrodes was 18 mm. At this time, the average (86.01 nm) and maximum (130.86 nm) Ra values were also optimal. Therefore, the best distance between the electrodes was 18 mm.

3.2.4 Optimization of the electrolyte temperature for the nitinol tube

According to Table 4, the fourth most important factor for the surface quality of the nitinol tubes was the polishing temperature. Therefore, a single-factor test was also performed for the electrolyte temperature. The parameter range of temperature was set to 14–36 °C. According to sections 3.2.1–3.2.3, polishing time was 9 min, voltage for polishing was 20 V, and the distance between electrodes was 18 mm. The Ra average fitting curve of the polished nitinol tube is shown in Figure 8.

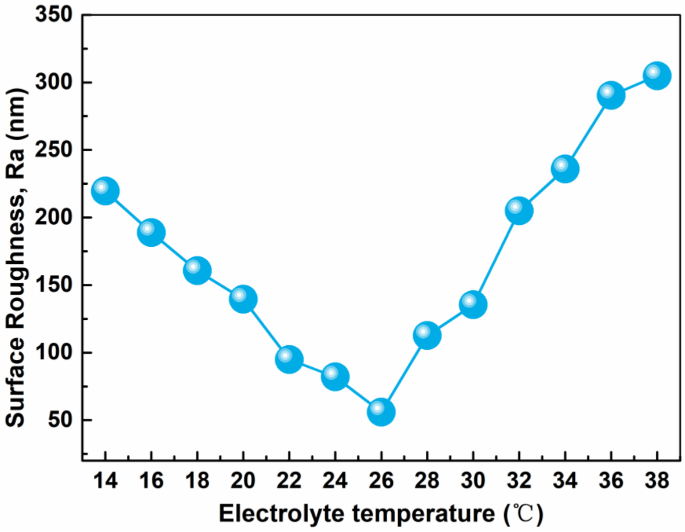

Figure 8. Polishing temperature (14-36 ℃) of the nitinol tube–Ra Average fitting curve

Figure 8 shows that the Ra first decreased and then increased as the polishing temperature increased. When the polishing temperature was 14 °C, the minimum, average, and maximum Ra values were 157.64, 219.52, and 291.54 nm. As the temperature increased, the Ra gradually decreased. When the polishing temperature reached 26 °C, the minimum, average, and maximum Ra values were 16.87, 55.96, and 90.87 nm. When the temperature continued to rise to 36 °C, the minimum, average, and maximum Ra values increased by 13.50-, 4.19-, and 2.57-fold, respectively. Therefore, the optimal polishing temperature was 26 °C.

3.3 Rate of reduction in wall thickness and the surface quality of the polished tube

3.3.1 Rate of reduction in wall thickness

As a biomedical material, the nitinol tube is widely used in the human body. Therefore, the change in the wall thickness of a nitinol tube after treatment should be studied. The best electropolishing parameters from section 3.2 were used to study the variation in the wall thickness with polishing time. To reduce the error in size, the size of the wall thickness was measured using an optical profiler. All results were expressed as the average of three measurements. The variation in the wall thickness with polishing time is shown in Figure 10. The wall thickness of the nitinol tube gradually decreased with the extension of the polishing time. After polishing for 9 min, the wall thickness was reduced by 42.32 µm, and the removal rate was 4.70 µm/min.

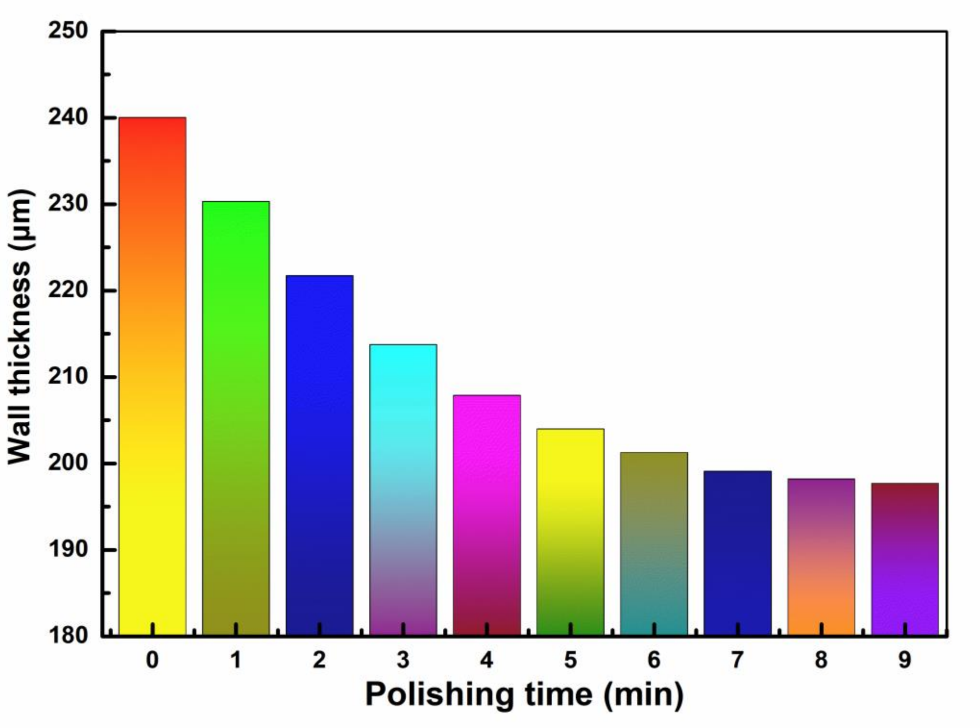

Figure 9. Variation in the nitinol tube wall thickness with polishing time (0-9 min)

3.3.2 Changes in the surface wettability of the tube

The wettability of nitinol tubes has an important influence on human tissues. The surface of the tube affects cell proliferation due to protein adhesion, which may affect the integration of the implant/tissue [32,33]. Therefore, the wettability of the surface is a key factor affecting the integrity of the implant material in the human environment. A contact-angle measuring instrument (OCA15EC) was used to measure the wettability. The surface of each tube was tested thrice, and the result was expressed as the average of measured values from the three experiments. The static contact angles before and after polishing (optimal electropolishing parameters) are shown in Table 5. Statistical analysis showed that the left, right, and average contact angles after polishing increased from 49.3°, 50.5°, and 49.9° to 83.6°, 82.8°, and 82.7°, respectively. Although the surface remained hydrophilic, its hydrophilicity was significantly reduced, probably because the surface quality of the tube was improved after polishing, and the adhesion of blood and protein was reduced [34]. Therefore, the biocompatibility of the nitinol tube was improved.

Table 5. Static contact angle before and after electrolytic polishing

	Left contact angle (°)	**Right contact angle (°)**	**Average contact angle (°)**
Before electropolishing	49.3	50.5	49.9
After electropolishing	82.6	82.8	82.7

3.3.3 Comparison of the morphologies of the nitinol tubes

Comparison of the visual image and morphology of the nitinol tube is shown in Figure 10. Figures 10(a) and 10(b) show the visual images of the nitinol tube before and after electropolishing, respectively. Many oxides were found on the surface of the tube before polishing, and the surface was rough and dim. The polished surface was bright and smooth. Figures 10(c) and 10(e) are magnified images of Figure 10(a). The prepolished surface flatness was very poor and had many oxides. Figures 10(d) and 10(f) are magnified images of Figure 10(b). The flatness of the polished surface was very high, and the surface quality had improved significantly.

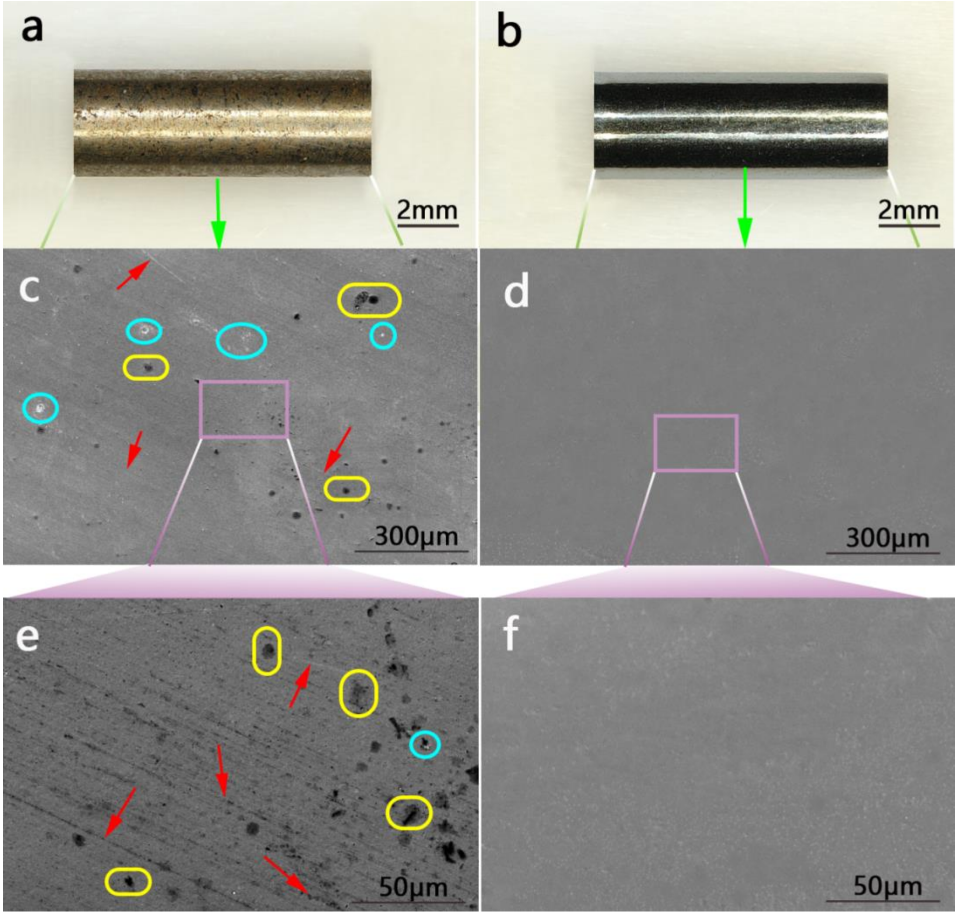

Figure 10. Microscopic morphology of the nitinol tube: visual images of the nitinol tube (a) before and (b) after electropolishing; (c) partially enlarged view of (a); (d) partially enlarged view of (b); (e) partially enlarged view of (c); (f) partially enlarged view of (d)

4. CONCLUSIONS

This study is the first to report the electropolishing of nitinol tubes by using an environment-friendly electrolyte. The electropolishing solution and parameters were optimized through orthogonal and single-factor experiments. The degrees of influence of the electropolishing parameters on the nitinol tube were investigated. The best combination of the process parameters was determined, and the surface quality of the tube was improved. The main conclusions were as follows:

(1) Adding 30 mL/L calcium chloride to 1 mol/L sodium chloride-glycol electrolyte (70% v/v) and ethanol mixture (30% v/v) significantly improved the polishing quality.

(2) Polishing time had the greatest influence on the quality of the polished nitinol tube, followed by voltage for polishing, distance between electrodes, and electrolyte temperature.

(3) Polishing time of 9 min, voltage for polishing of 21 V, distance between electrodes of 18 mm, and electrolyte temperature of 26 °C resulted in the best Ra of 16.87 nm.

(4) Polishing with the best electropolishing parameters resulted in reduced wall thickness by 42.32 μm and removal rate of 4.70 μm/min. The average hydrostatic contact angle increased by 32.8°, which improved the biocompatibility.

ACKNOWLEDGEMENTS

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